USED LUBE OIL ANALYSIS &

ANALYTICAL FERROGRAPHY

Paul Goldman MRT Laboratories 305 Nebraska Ave South Houston, TX 77587

713-944-8381

Oil Analysis Provides Four Important Pieces of Information

- 1) Condition of Equipment Lubricated Components (Bearings, gears, cylinders, & other lubricated components)
- 2) Condition of Lubricant -- Can we continue to use the lubricant with a high level of confidence?
- 3) Level of Contamination -- How contaminated is the lubricant? What is the contamination? Where did it come from? How can we prevent it from occurring again?
- 4) What do I do next?

Condition of Lubricated Components

Determined primarily by:

Spectroscopy for ionic and small metal particles

Direct Read Ferrography for mostly ferro-magnetic particles

-- or --

PQ Index for ferro-magnetic particles

ISO Particle Count for all particles according to size range.

Spectrographic Metals Analysis

1: Atomic Emission Rotrode Spectrometer

Particle size limitation $\sim 7~\mu$, depending on metal and level of surface oxidation

Accurate to about ± 5%

No dilution of sample is required

Results include wear, additive, and contaminant metals in parts per million (ppm)

Spectrographic Analysis

2: Inductively Coupled Plasma Spectrometer

Particle size limitation ~ 7 μ, depending on metal

Accurate to about + 1%

Dilution of sample is required

Results include wear, additive, and contaminant metals in parts per million (ppm)

Usually 18 to 22 wear, additive, and contaminant metals are detected

Wear

Iron

Copper

Lead

Tin

Chromium

Titanium

Nickel

Aluminum

Silver

Cadmium

Additive

Zinc

Phosphorus

Magnesium

Calcium

Barium

Molybdenum

Antimony

Contaminant

Boron

Silicon

Potassium

Sodium

Vanadium

Metals by Atomic Emission Spectroscopy

		o 🕜Do	vvn 🚮 Tok		Next	5	0	Previo	use 5			
I	Sam	ple Inform	nation				W	ear Me	tals			
ı	•	Hrs/Miles	Samp Date	Iron	Сорре	ŧTin	Lead	Chrom	Nicke	Alumi	Titan	Silv
ı	5033004		2005-03-01	38	18	D	D	0	D	0	O	0
ı	5023004		2005-02-01	24	11	D	D	O	Ō	O	0	0
ı	5013004		2005-01-01	18	5	0	O	0	0	D	D	0
ı	4123004		2004-12-01	16	3	0	O	0	0	D	D	0
ı	4113004		2004-11-01	10	1	D	O	0	O	0	0	0
ı	4103004		2004-10-01	12	3	D	D	0	0	O	O	0
ı	V	Vateh Adviso	orv	15	10	10	10	5	5	10	5	- 5
I	W0	30	20	20	20	10	10	20	10	10		
		Reference	-	0	0	0	0	0	0	0	0	0

Basic bearing, gear, and shaft wear metals are monitored and trended

Condition of Lubricated Equipment Components Metals by Atomic Emission Spectroscopy

	i	Addit	tive N	letals	Contaminant Metals						
Calcil	Magne	Zinc	Phosp	Silic Sodiu Boron Potas Vanad							
0	0	0	0	0	0	0	15	82	0	44	0
0	0	0	0	0	0	0	8	38	0	24	0
0	0	0	0	0	0	0	4	18	0	11	0
0	0	0	0	0	0	0	0	4	0	6	0
0	0	0	0	0	0	0	0	1	0	2	0
0	0	0	0	0	0	0	0	1	0	0	0
							10	40	10	15	10
							20	40	20	30	20
0	0	0	0	0	0	0	0	0	0	0	0

Additive metals are monitored.

Contaminant metals will help indicate source of contamination

Ref: MRT Laboratories

Direct Read Ferrography

Reported as Direct Read Small (DRS) & Direct Read Large (DRL)

Unit-less numbers range from 0.1 to 180.0

Indicative of ferromagnetic particles & some nonmagnetic particles

Can "see" large particulates (> 300 microns)

Trico's DR7 Direct Read Ferrography Instrument



Sample flows through a glass tube sitting on an inclined magnet with two light paths – DRL and DRS.

The attenuation of light in each light path during the run produces a unitless result ranging from 0.00 to 180.00

Ferro-magnetic particles as well as heavier non magnetic particle contribute to the light attenuation.



Analex's PQL Ferrous Debris Monitor

A magnetometer that measures the mass of ferro-magnetic debris in a sample and displays this as a PQ (Particle Quantifier) index.

The PQ Index is a quantitative unitless number ranging from 0 to 5000

Independent of particle size

Particle Count Analysis



Determines Cleanliness of Lubricant and Other Fluids by measuring scattered laser light.

Reported at 4, 6, 14 micron thresholds (ISO 4406.1999)

ISO Code

Expressed as x/x/x

Ex: 16/14/11

$$16 - > 4 \mu$$

$$14 - > 6 \mu$$

$$11 - > 14 \mu$$

Number of Particles Per Milliliter

100 4400 D N 1 1 1	O Th	Lasa Than
ISO 4406 Range Number	Greater Than	Less Than
24	80,000	160,000
23	40,000	80,000
22	20,000	40,000
21	10,000	20,000
20	5,000	10,000
19	2,500	5,000
18	1,300	2,500
17	640	1,300
16	320	640
15	160	320
14	80	160
13	40	80
12	20	40
11	10	20
10	5	10
9	2.5	5
8	1.3	2.5
7	0.64	1.3
6	0.32	0.64
5	0.16	0.32
4	0.08	0.16
3	0.04	0.08
2	0.02	0.04
1	0.01	0.02

Particle Count Analysis

Sample	Information					Physical and Other Tests							
Sample No	Samp Date	V@40c	AN	Flash	ISO Code	KF	Color	4u	6u	14u	21u	38u	68u
5033005	2005-03-01	26.5	0.09	295	20/17/13	62	3.5	8716.5	1108.8	72.6	14.3	3.5	2.2
5023005	2005-02-01	29.1	0.07	350	7/14/12	42	2	1023.9	159	38.4	8.9	2.3	0.6
5013005	2005-01-01	31.9	0.06	370	18/14/12	48	2	544.4	110.7	30.6	6.6	1.9	0
4123005	2004-12-01	31.7	0.04	285	15/14/12	58	2	303.7	85.6	32.1	2.6	0.5	0
4113005	2004-11-01	32.2	0.04	385	16/13/12	66	2	488.5	74.3	36.8	3.6	0.4	0
4103005	2004-10-01	32.1	0.05	380	16/13/11	51	2	467.2	59.4	18.2	6.4	1.6	0
Watch	n Advisory	28.8-35.2	0.15	360	20/18/16	100	2						
Warnin	Warning Advisory		0.2	340	21/19/17	200	4	/					
Ref	Reference		0.05	395	13/11/07	43	0	41.6	13.5	1.3	0.5	0	0

ISO 4406 Code plus actual Particles per CC of oil reported.

The Need for Microscopic Particle Examination

From Standard Lube Oil Analysis results:

We know there are metals present

	Wear Metals											
Iron	Сорре	≅Ti⊓	Lead	Chrom	Nicke	Alumi	Titan	Silv				
38	18	D	D	O	D	0	Ō	O				
24	11	O	0	Ō	O	0	0	0				
18	5	O	0	O	D	0	O	0				
16	3	0	O	Ō	D	0	0	0				
10	1	0	0	D	D	0	D	0				
12	3	0	0	0	0	0	Û	0				

We know there are particles present

4u 8716.5	6u 1108.8	14u 72.6	21u 14.3	38u 3.5	68u 2.2
1023.9	159	36.4	8.9	2.3	0.6
544.4	110.7	30.6	6.6	1.9	0
303.7	85.6	32.1	2.6	0.5	0
488.5	74.3	36.8	3.6	0.4	0
467.2	59.4	18.2	6.4	1.6	0

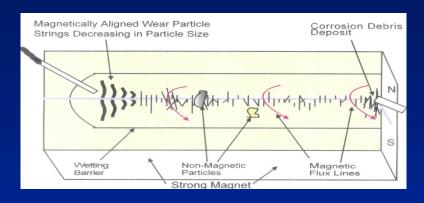
..but were not sure what component they came from or what's causing them to be generated...

Microscopic Particle Analysis is usually performed as a reaction to routine test results.

Often performed for cause, such as increased bearing temperature, increase in filter DP, etc...

Best way to identify particulates reported by the ISO 4406 particle count, PQ Index, or Direct Read Ferrography

Best way to evaluate wear severity and wear mode



Ferrogram is made on Slide Maker



Slide is washed with heptane that has been triple filtered through a .45 micron filter. This removes oil residue.

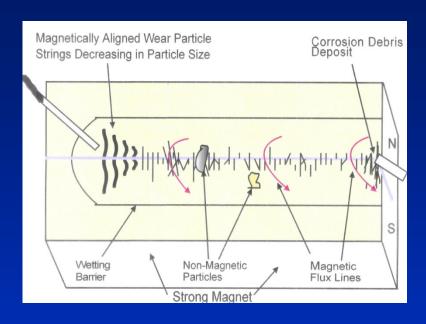


Slide is visually examined using a bi-chromatic optical microscope.

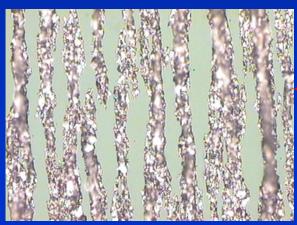
Lenses provide 100X, 500X, and 1000X views.

Color filters and polarizers help the analyst identify particles.

Heating the slide to transition temperatures will aid the analyst in identifying particles and determining broad metallurgy categories.



Analyst looks at magnetic alignment of particles to determine which are ferromagnetic and which are other material.

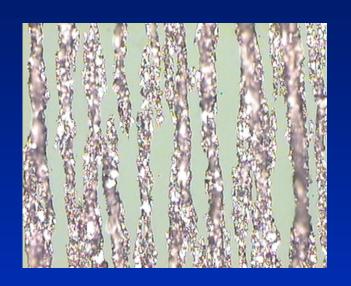


Metallic particles in magnetically aligned strings, indicating iron or steel.

PARTICLE TYPES

- Normal Rubbing Wear
- Severe Sliding Wear
- Cutting Wear
- Bearing & Gear Wear
- Spheres from Rolling Element Fatigue
- Black Oxides from Lubricant Starvation
- Babbitt Particles

- Corrosive Debris
- Lube Degradation
- Varnish & Lacquer Particles
- Sand & Dirt
- Fibers
- Contaminant Spheres
- Red Oxides from Water
- Red Oxides from Fretting Corrosion.



NORMAL RUBBING WEAR

Individual particles are generally 5 microns and below. The quantity of these particles determines the wear rate.



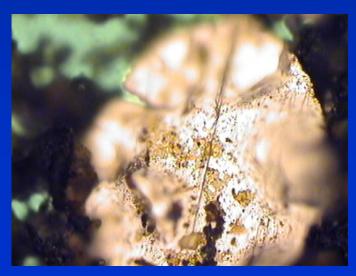
SMALL CUTTING WEAR

Small cutting wear particles such as this are usually caused by abrasion wear due to contaminants or other wear particles in the lubricant



LARGE CUTTING WEAR

Large, curled cutting wear particles such as this are usually generated as a result of misalignment or abrasive particle embedded in a Babbitt bearing.



2013 MRT Laboratories, LLC

LARGE LAMINAR PARTICLES

Large, rounded, flat particles with a width/thickness ration of 20-1 are generated as a result of rolling element fatigue. Macro-spalling is indicated if the particles are in the 40 micron size range.

Ref: MRT Laboratories



FATIGUE CRACK SPHERES

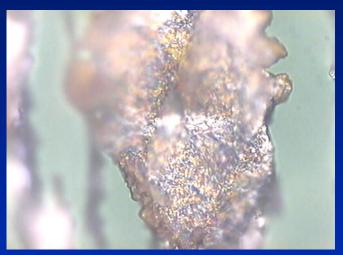
Fatigue cracks can generate small 1-10 micron spherical particles, sometimes copious amounts.

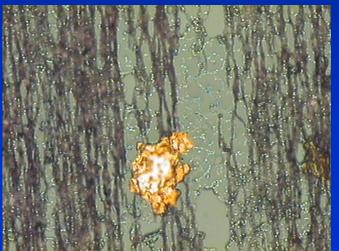


GEAR ROOT OR TIP WEAR

Severe Sliding Wear

Gear root or tip wear generates long, flat particles, often with striation marks as a result of the sliding that occurred during generation.





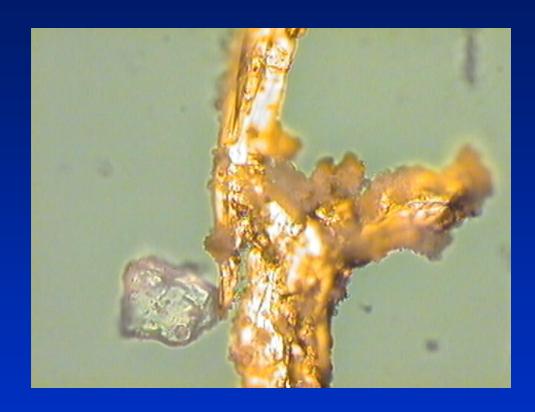
BABBITT PARTICLES

Babbitt particles can be identified by their stippled, multicolored surfaces.

Their edges will often melt slightly when heated to 625° F

COPPER ALLOYS

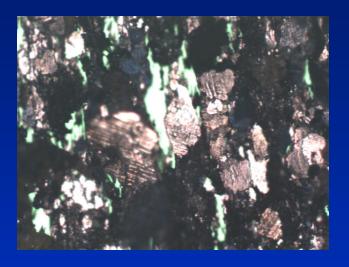
Unheated, yellow metals are easily identified by their color. These are usually generated at roller bearing cages as a result of a lubrication problem.



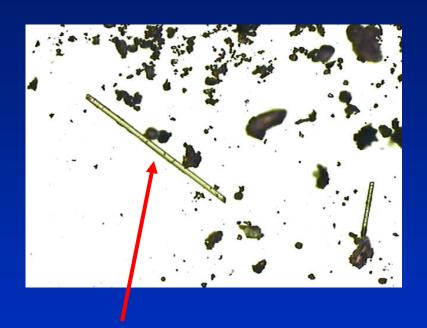
The tortuous shape of the particle will help determine the severity of wear.



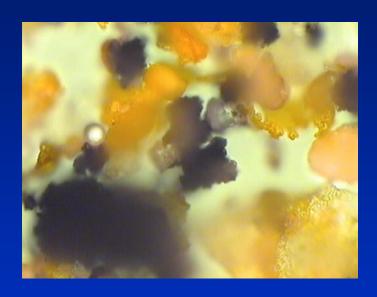
Corrosive Wear Aged or Oxidized Lubricant



Black Oxides & Gear Wear Lubricant Starvation







Large Red Iron Oxides from water contamination

Contamination

Airborne or Waterborne contaminants





Filter Degradation

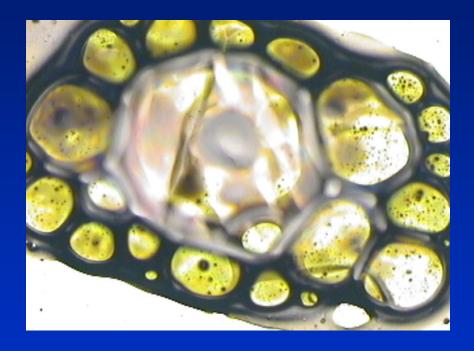
Varnish and Lacquer Particles





Product Contamination

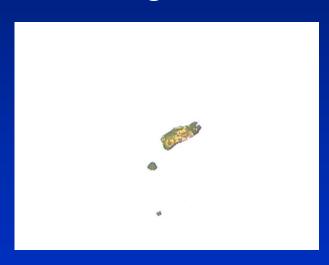
Contamination



We haven't a clue

Individual Particles can be examined usually at:

100X Magnification



At 100X, it can be determined this 500 micron particle is significant

500X Magnification



At 500X, details of the particle and its morphology become more evident.

1000X Magnification



At 1000X, minute details of the particle can be observed.

1000X Magnification



A measuring device on the microscope allows us to focus on the top and bottom of the particle and determine the 'thickness' in microns



At 1000X, particles as small as 1-2 microns can be observed and most often identified

2 micron ferrous wear particle.

Temper Colors

Temper Colors

Determination of Metallurgy

TRANSITION TEMPERATURES

500F - Organic material will char, shrink and/or shrivel.

625F - Carbon Steel will turn blue

High Alloy steel will remain white

Cast iron and medium alloy steel will turn straw colored

Babbitt surface will oxidize and obtain a stippled, multicolored appearance. Edges may melt slightly.

Aluminum and Chromium will remain white

Copper/brass/bronze will turn dark bronze with streaks of blue, red, and purple, depending on the alloy

Temper Colors Determination of Metallurgy

TRANSITION TEMPERATURES – 750 Deg F.

Carbon steel ➤ Light gray – light straw Cast iron and medium alloy steel —— Deep bronze High alloy and Stainless Steel → No change or slight yellowing **Aluminum & Chromium →** No change Further stippled surface and some **Babbitt** edge melting **Copper Alloys ——→** Deep straw with red, purple, and blue coloring depending on the alloy **Organics** Further charring, shrinking, or vaporization

Temper Colors

Determination of Metallurgy

TRANSITION TEMPERATURES - 900 Deg F.

Carbon steel Dark gray – dark straw Cast iron and medium alloy steel ----Deep bronze with mottled bluing High nickel alloy steel **Bronze with significant bluing** Stainless Steel Light straw to bronze, some stainless may have slight bluing **Aluminum & Chromium** No change **Babbitt** Surface completely oxidized dark. **Edges melted. Copper Alloys** Dark straw, may still have slight amount of reds, purples, and blue, depending on the alloy **Organics** Further charring, shrinking, or vaporization.

Temper Colors

Determination of Metallurgy

TRANSITION TEMPERATURES - 1000+ Deg F.

Carbon steel

Cast iron and medium alloy steel

Deep bronze with heavy bluing

High nickel alloy steel

Blue or blue/gray

Stainless Steel

Darker bronze with heavier mottled bluing

Aluminum & Chromium

No change

Babbitt

Surface completely oxidized dark. Edges definitely melted.

Copper Alloys

Dark straw, little reds, purples, and blue,

Organics — Mostly melted into blobs or heavily deformed, or completely vaporized

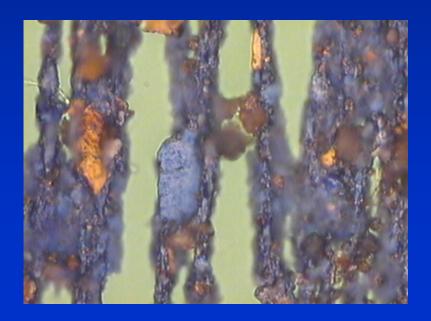
depending on the alloy

Temper Colors Example: Determination of Metallurgy

Heated to 625F.

Those carbon steel particles <~100u will turn bluish when heated to 625F for 90 secs.

Much larger carbon steel particles will only turn slightly blue unless heated for a longer period of time.

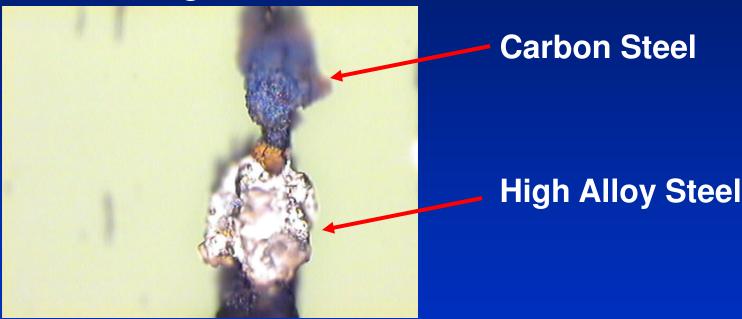




Temper Colors

Example: Determination of Metallurgy

After heating slide to 625F



If an excessive number of both these type of steel are present and the sample is from rolling element bearings, it probably indicates a loose fit.

Temper Colors

Example: Determination of Metallurgy

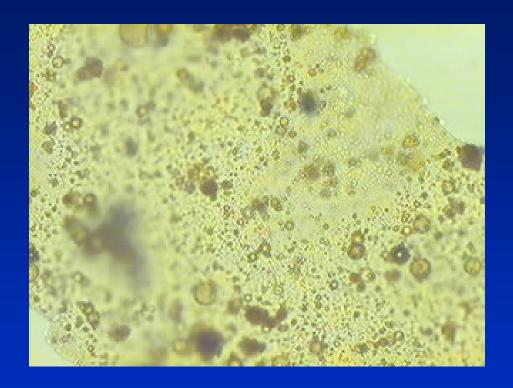


Babbitt will have a stippled appearance, along with often times melted edges when heated to 625F and above.

Other Determinations as a Result of Heating the Slide



Rust particles will turn bright reddish/orange as they are heated. Heating will have no effect on fiberglass debris from filters. This is one method of identifying fibrous material.



Friction polymers in a gear oil, indicating moderate to heavy lubricant stresses.



O-ring or Gasket Material

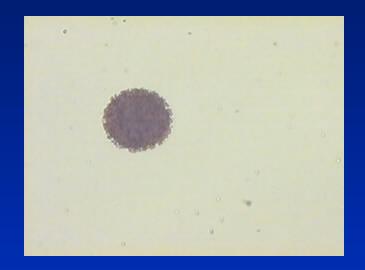


500X – heavy spalling from roller bearing element. When heated to 625f for 120 seconds, these turned blue, indicating carbon steel.



100X – Wear from a slinger ring.





Plastic Dust from filter or product.

After heating to 625 F, particle melts.

Other Identifiable Particulates

Catalyst Fines

Sandblasting Sand

Molybdenum Disulfide Additives

Graphite Additives

Teflon Tape particles

Wax Globules

Bio-Mass

Insect Parts

Microscopic Particle Analysis

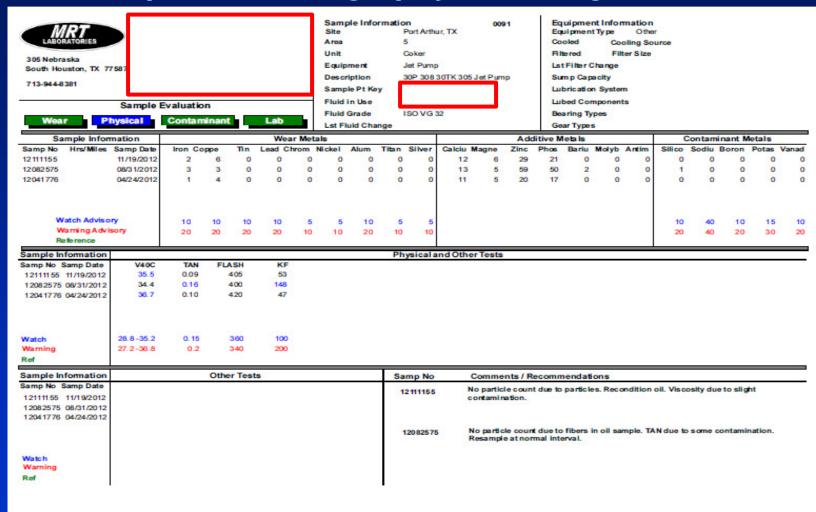
Additional Points!!

Often what is NOT observed is as important as what is observed

When used in conjunction with routine Lube Oil Analysis, Analytical Ferrography will enhance the effectiveness of your Analysis Program

The more experienced the ferrographer, the better the results, and the more familiar the ferrographer is with the equipment in question, the better the results.

Share information with your lab. When a piece of equipment is opened for inspection, let your lab know what you found compared to the analysis report.



Nothing alarming in standard report, but.....



South Houston, TX 77587

713-944-8381



Sample Point Information

Site PortArthur, TX

Unit Coker Equipment Jet Pum

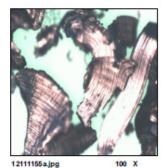
Description 30P 308 30TK 305 Jet Pump

Sample Pt Key

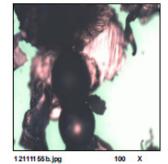
-- Page 2 - IMAGE REPORT

Sample Information

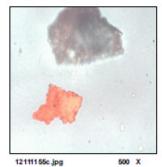
Sample Number 12111155 Sample Date 11/19/2012 Report Date 11/20/2012



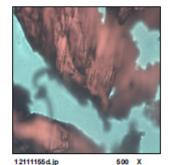
These 200 to 500 micron ferrous cutting wear particles are typical of the others on the slide. These are abrasive wear. Check unit for noise or vibration. Recondition oil.



These two 160 micron metal spheres are the largest of the few seen. These are from bearing fatigue



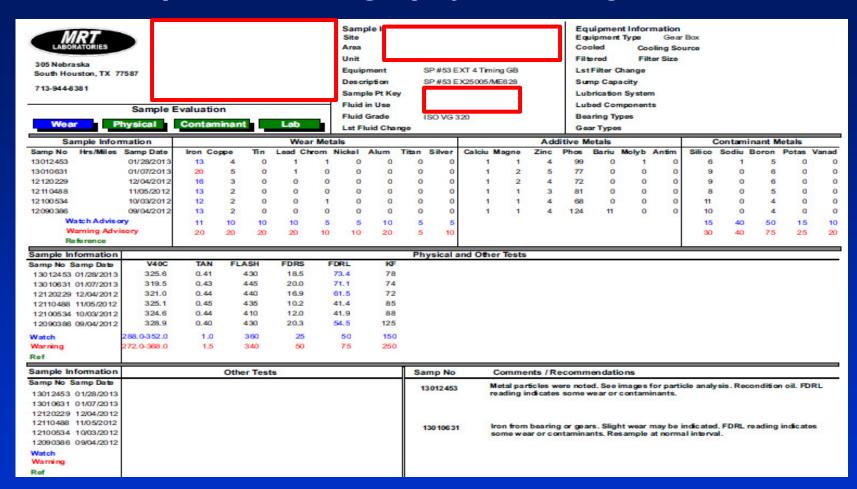
This 40 micron copper particle is one of 2 seen. The other particle is filter material



This is after heating the slide to 625F. Most of the ferrous particles turned bronze. They are medium alloy steel. A few turned blue, carbon steel. Most of these appear to be from the bearing.

Several large ferrous cutting wear particles (abrasive wear) were seen. Most of these appear to be from the bearing. Check for noise or vibration. Recondition oil. A few large metal spheres that appear to be from bearing fatigue cracks were also seen.

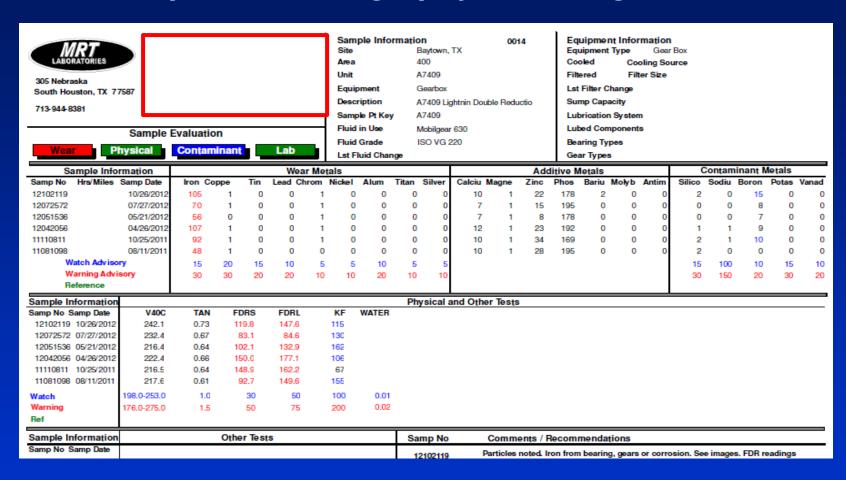
Severe wear was detected with ferrography



Steady iron increase, then drop while FDRL increases



This is what an extruder gearbox looks like to us when it is starting to fall apart!



Just changing the oil doesn't always solve a wear problem.



305 Nebraska

South Houston, TX 77587

713-944-8381



Sample Point Information

0014

Baytown, TX

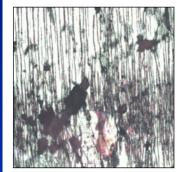
Area 400 A7409 Gearbox

Description A7409 Lightnin Double Reductio

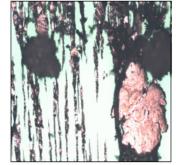
Sample Pt Key A7409 -- Page 2 -- IMAGE REPORT

Sample Information

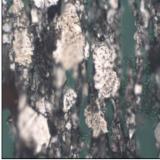
Sample Number Sample Date 10/26/2012 Report Date 10/31/2012



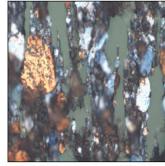
Many fibers were seen. Some type of clear non-metallic material was also seen (clear particle at bottom). This particle melted when heated. It could be product. Many ferrous corrosion particles were seen.



This 300 micron ferrous wear particle appears to be from a bearing. It was the largest seen. Most of the many wear particles were 30 microns or less. Many were 30 to 80 microns. Check for noise or vibration. Some severe wear may be indicated.



These are typical of the ferrous wear particles. Most appear to be gear wear. A few ferrous cutting wear particles are also seen. Recondition



This is after heating the slide to 625F. Many of the ferrous particles tunred blue. They are carbon steel. The bronze color particles are medium alloy steel.

If the wear mode is other than contaminated lubricant the wear process will continue even after changing the oil

QUESTIONS?

Thank You!!